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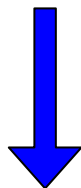
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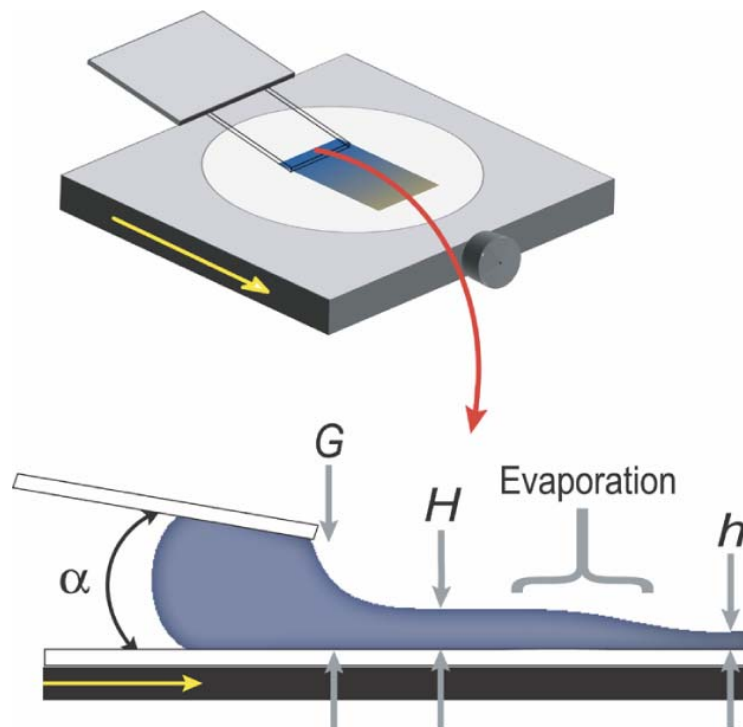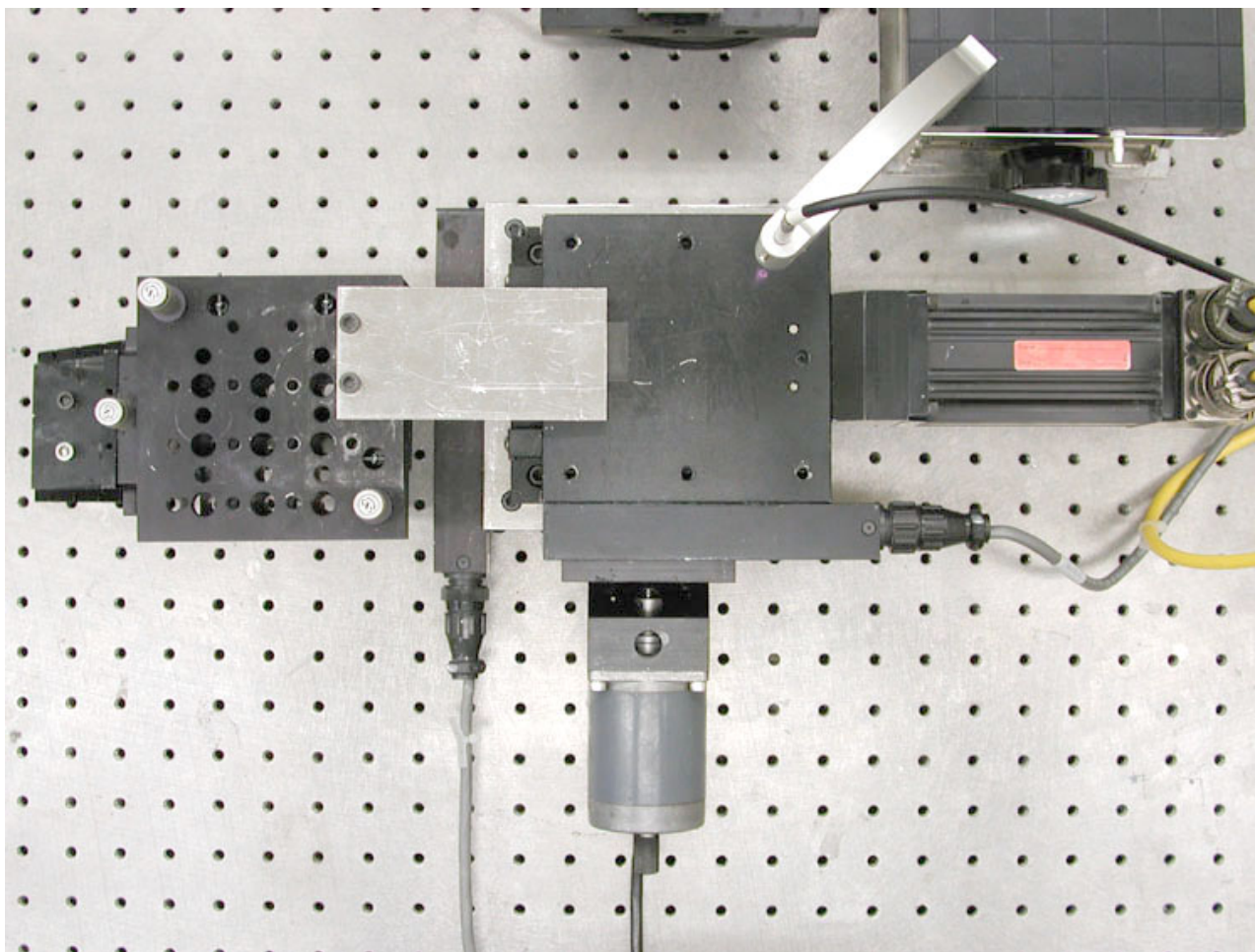
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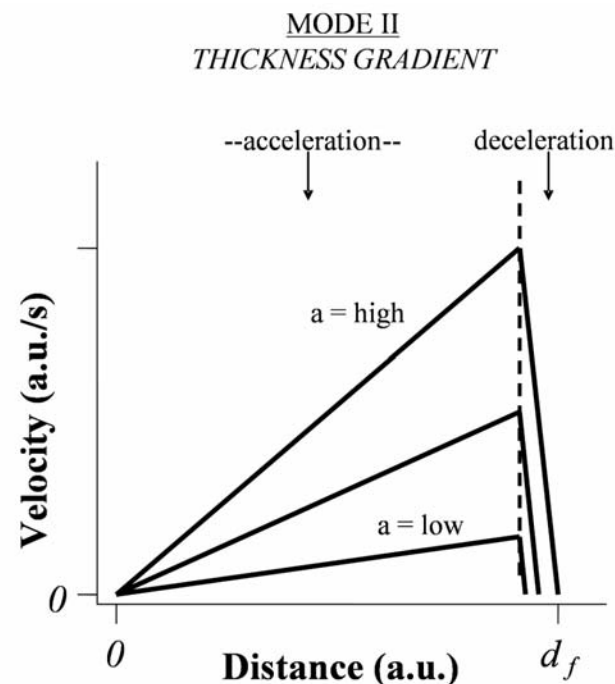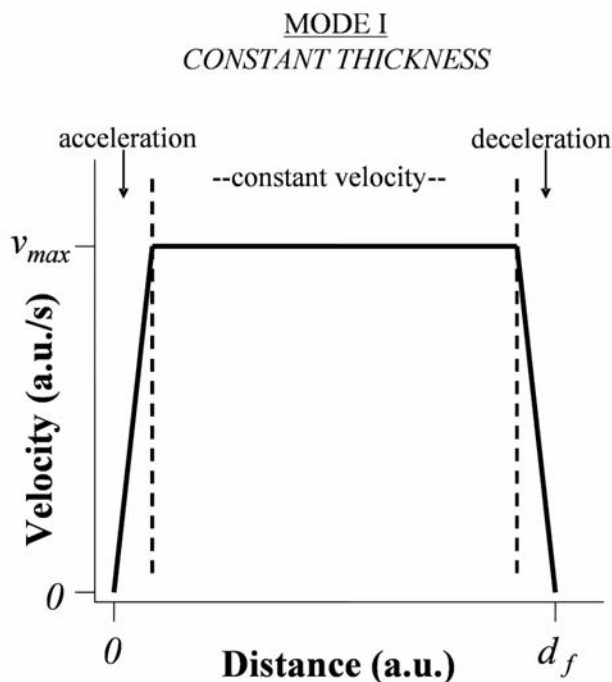
NIST Gradient Flow Coater

Christopher M. Stafford, _Kristen E. Roskov, Thomas H. Epps III, Michael J. Fasolka_

Polymers Division
Materials Science & Engineering Laboratory
National Institute of Standards and Technology

Why is thickness so important?

- ❑ Film thickness is a critical parameter in many applications
  - ❑ self-assembly of block copolymers
  - ❑ film stability / dewetting
  - ❑ pressure sensitive adhesives
  - ❑ fundamental polymer physics
  - ❑ barrier coatings
  - ❑ photoresists / imprint resists
  - ❑ electronic packaging
  - ❑ advanced coatings

- ❑ Offers a rich parameter space to probe material properties and response surfaces

- ❑ NIST flow coater opens the window to combinatorial libraries of film thickness in the range of nm to μm

Flow coating – basics

- ❑ Capillary forces hold polymer solution between a stationary knife blade and a substrate
- ❑ Frictional drag is exerted on the solution as blade accelerates over substrate

Low v -> capillary forces win
thinner films

High v -> drag wins
thicker films

Acceleration yields thickness gradient

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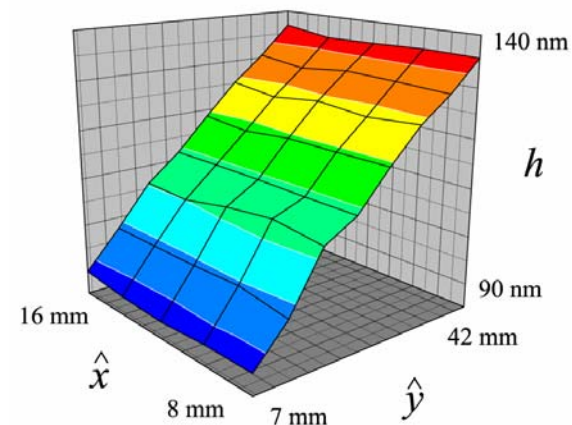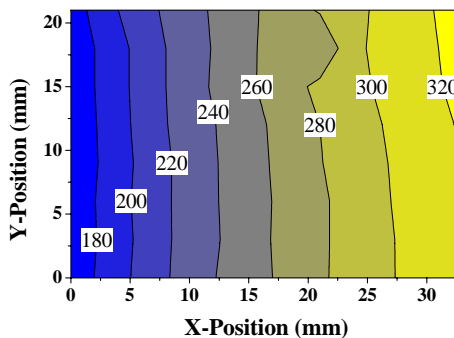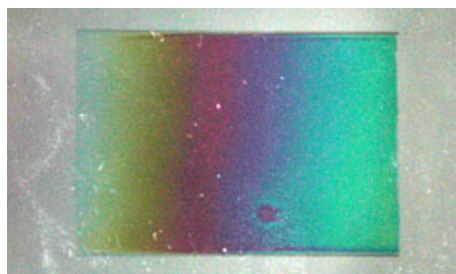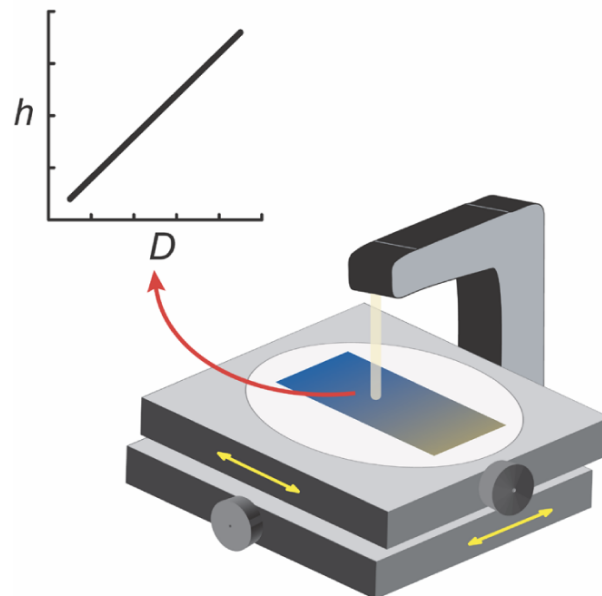
Flow coating - in action

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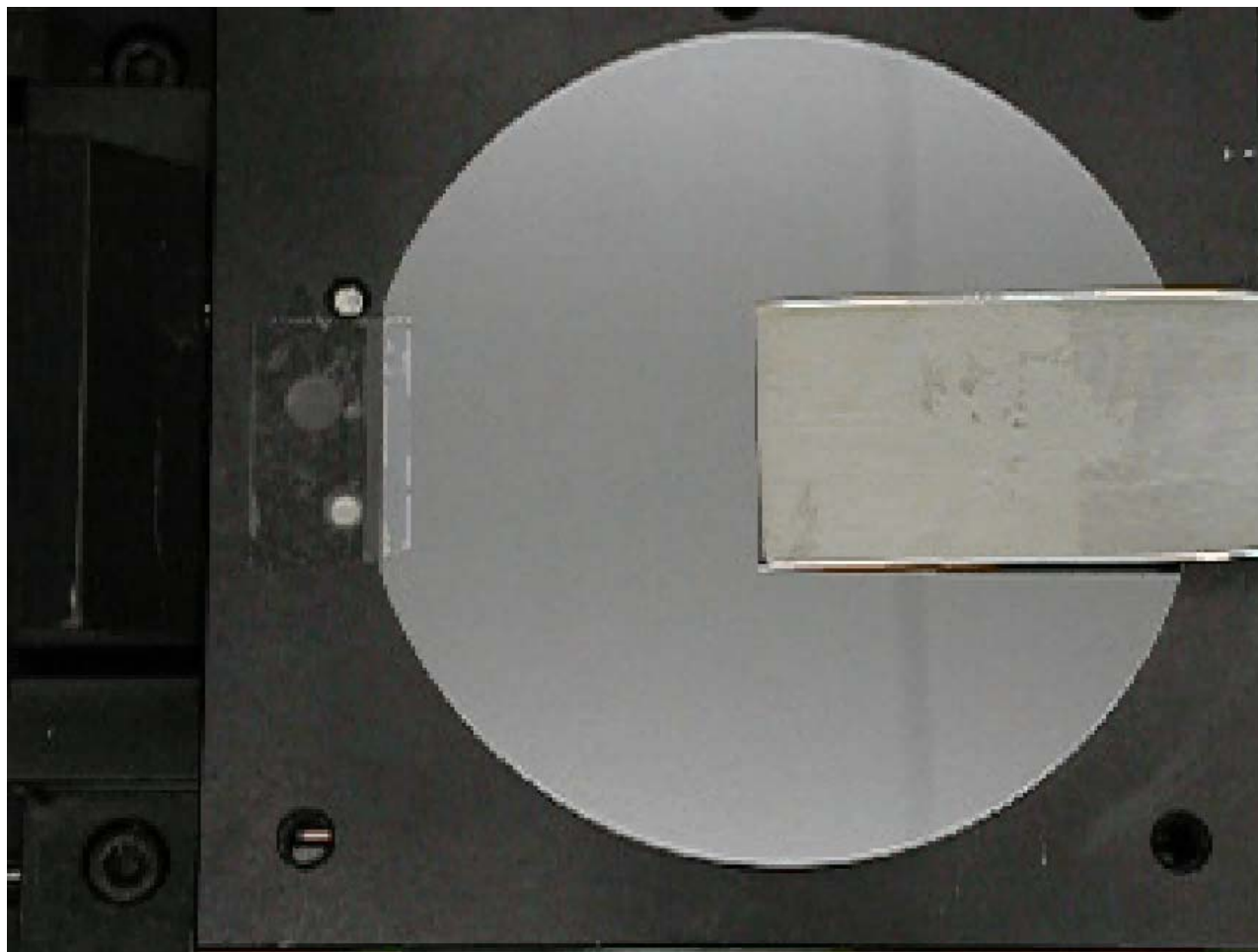
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❑ **Mode 1**: Uniform Film Thickness
  ❑ Acceleration is set very high
  ❑ Thickness dictated by maximum stage velocity (input v_{max})

❑ **Mode 2**: Thickness Gradient Films
  ❑ Acceleration is set low
  ❑ Thickness dictated by instantaneous velocity (controlled by a)

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- Measurement – thickness
- Thickness measured via:
  - Interferometry
  - Ellipsometry
  - Profilometry
- Visualization
  - Contour plots
  - Surface plots

Stafford et al. *Rev. Sci. Instrum.* **2006**, *77*, 023908.

Mode I – constant thickness

Thickness is linear with velocity. Good start!

Since thickness is linear with velocity, gradient should also be linear with distance (instantaneous velocity).

Legend:
- $a = 10$ mm s^{-2}
- $a = 8$ mm s^{-2}
- $a = 6$ mm s^{-2}
- $a = 4$ mm s^{-2}
- $a = 2$ mm s^{-2}

h (nm) vs d (mm)
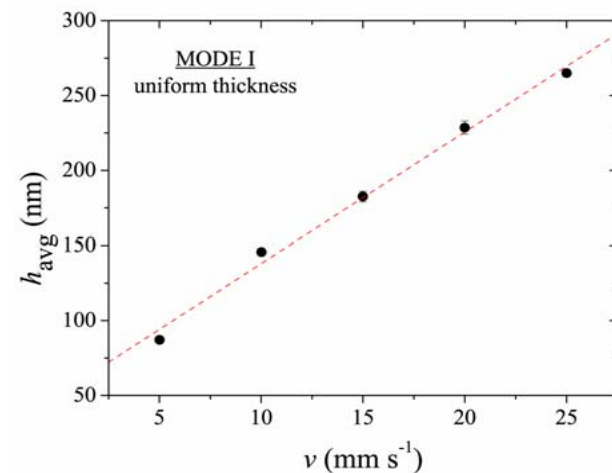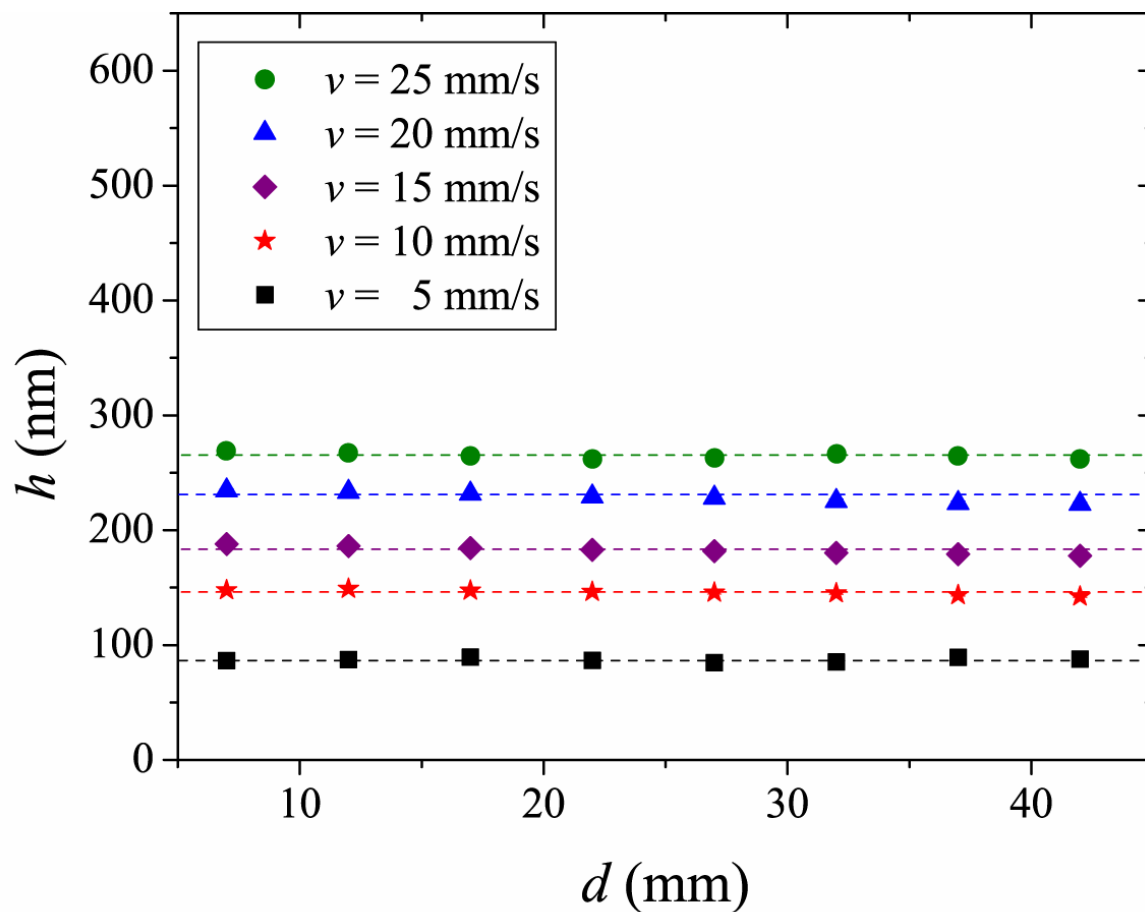
MODE II
thickness gradient

$\Delta h / \Delta d$ (nm/mm) vs a (mm s^{-2})

Thickness gradient can go from 10s of nanometers to several microns.
We have not explored the upper limit to the technique yet.
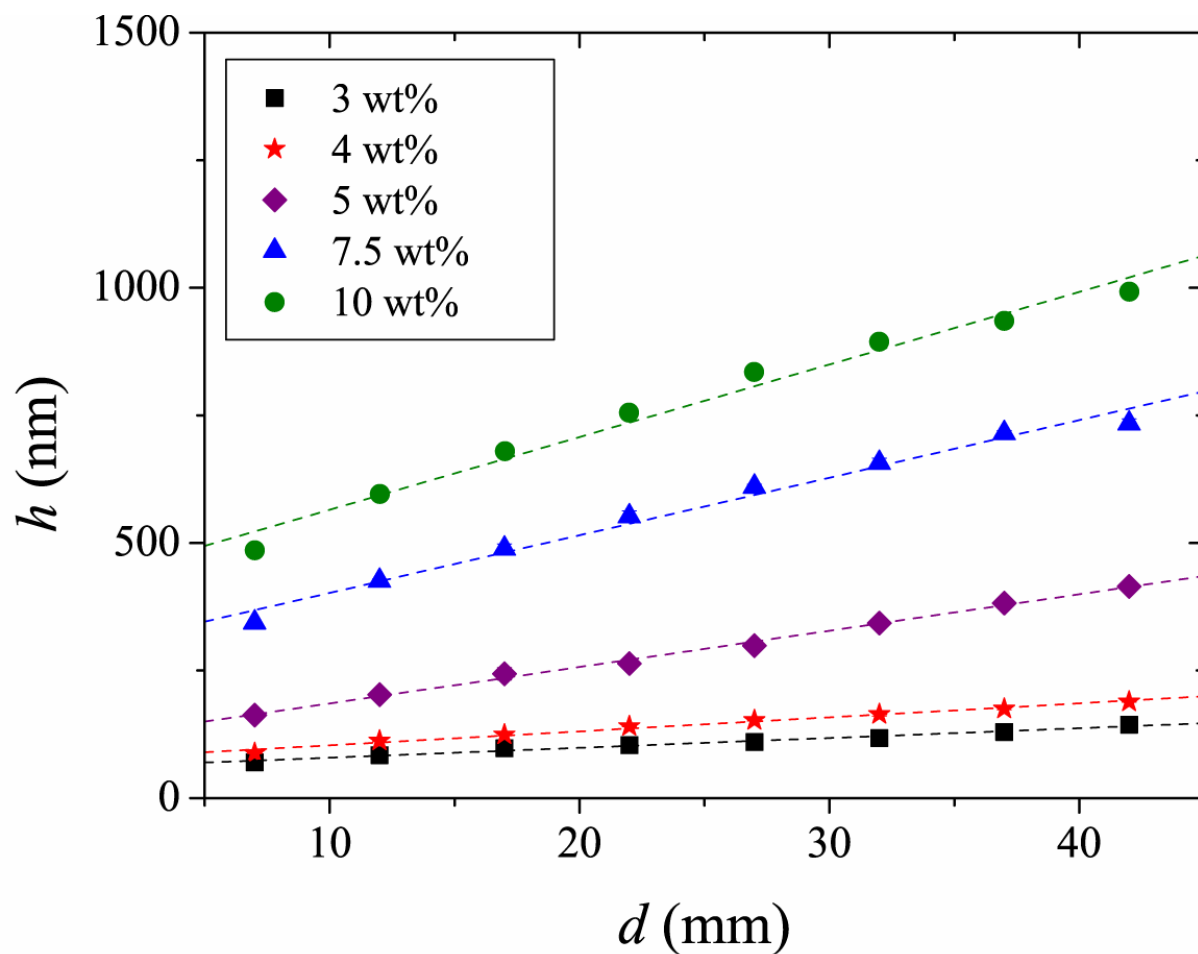
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solvent	b.p. (^{o}C)	v. p. @ 20 ^{o}C (mm Hg)	ρ (g mL^{-1})	η @ 20 ^{o}C (cP)
Chloroform	60.5 - 61.5	160	1.492	0.58
Toluene	110 - 111	22	0.865	0.59
PGMEA	145 - 146	3.7	0.970	1.31

◀— Drying time on the order of minutes

◀— Drying time on the order of seconds

All solutions had same wt% polymer.

Solution viscosity very different!!

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Applications – film stability
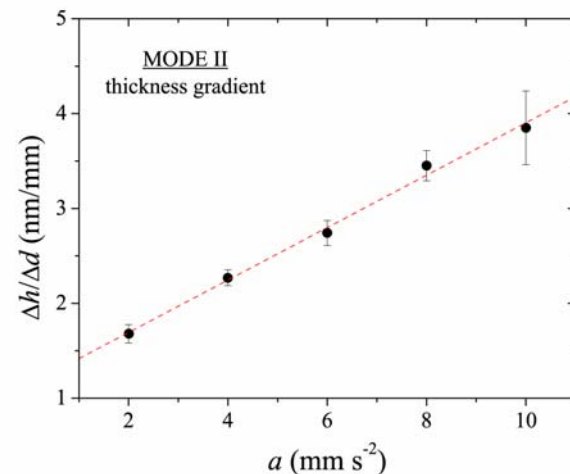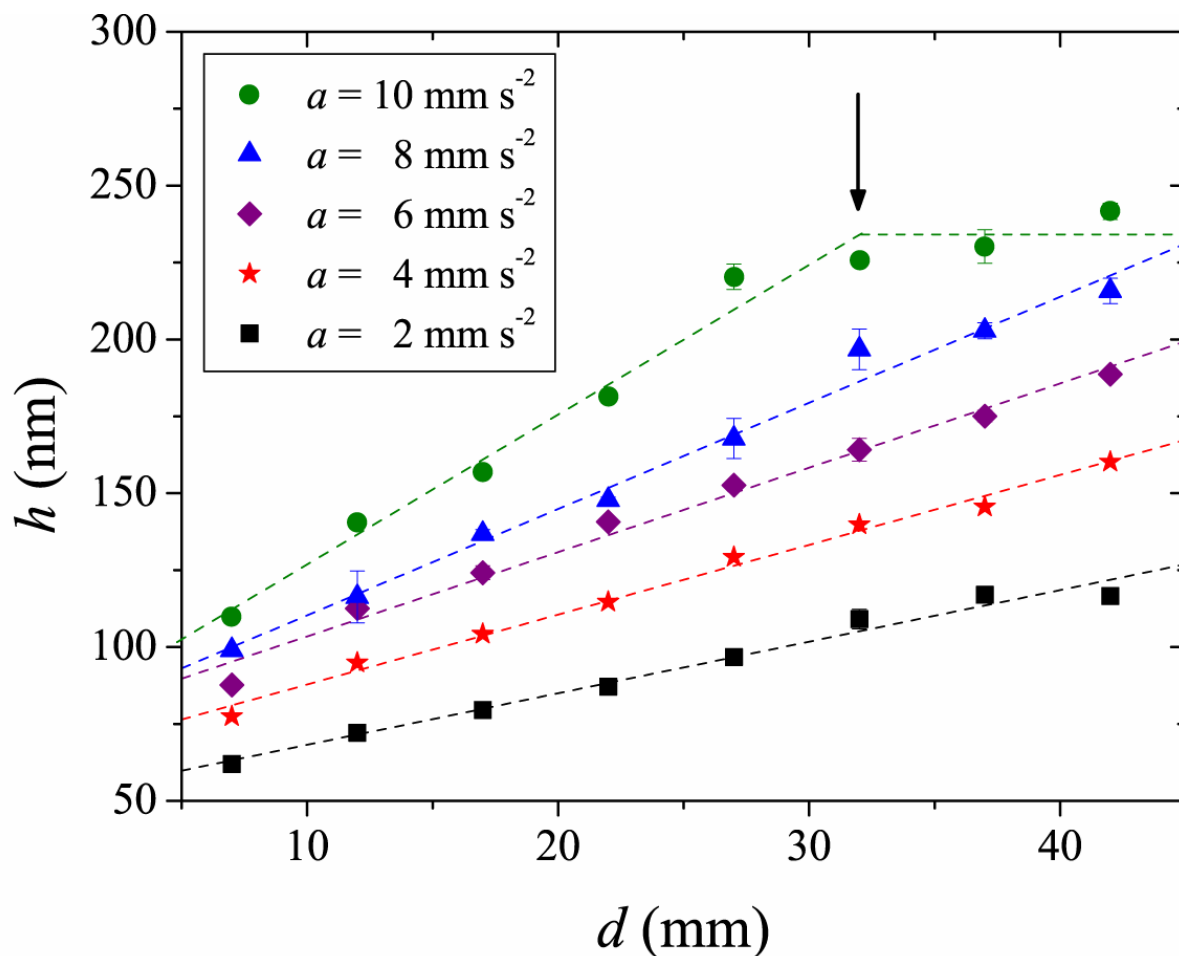
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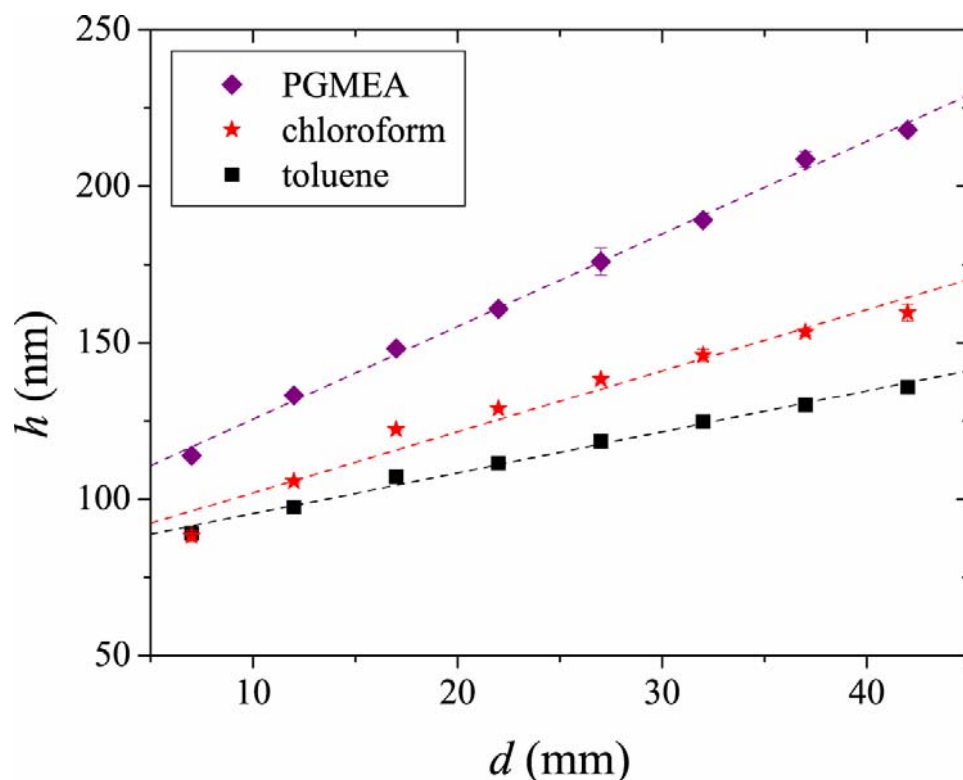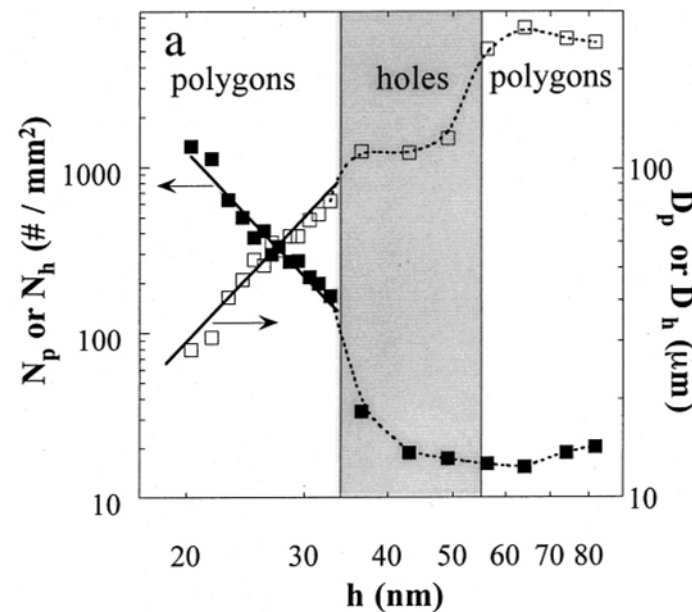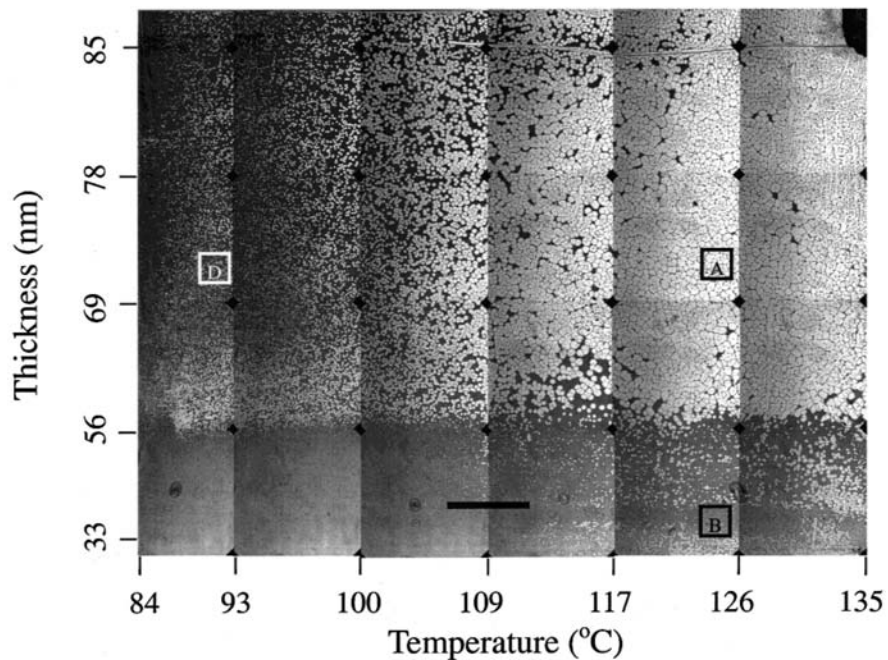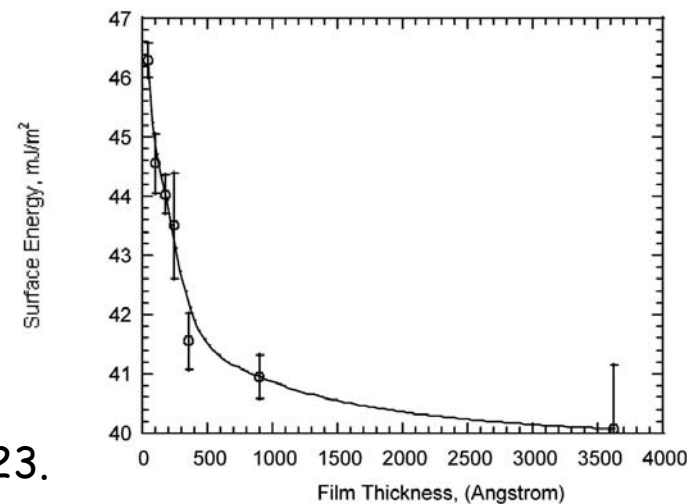
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Meredith et al. *Macromolecules* **2000**, *33*, 9747-9756.

Ashley et al. *Langmuir* **2005**, *21*, 9518-9523.

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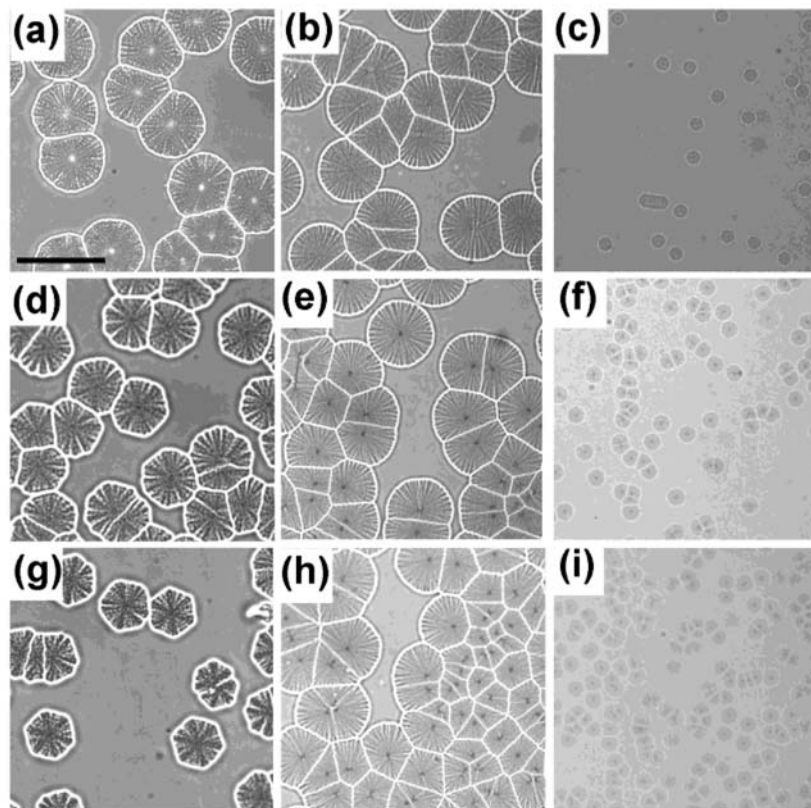
Beers et al. *Langmuir* **2003**, *19*, 3935-3940.

(154 ± 2) nm (140 ± 2) nm (135 ± 2) nm (108 ± 2) nm

b. 125 °C   120 °C   116 °C   111 °C
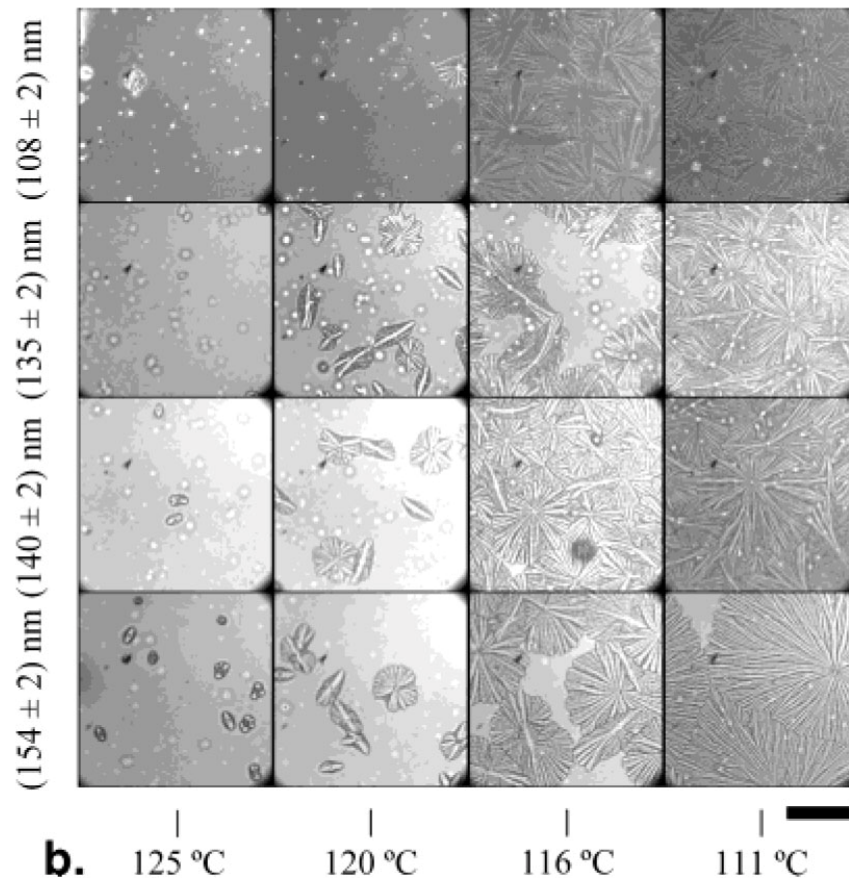
Walker et al. *Langmuir* **2003**, *19*, 6582-6585.

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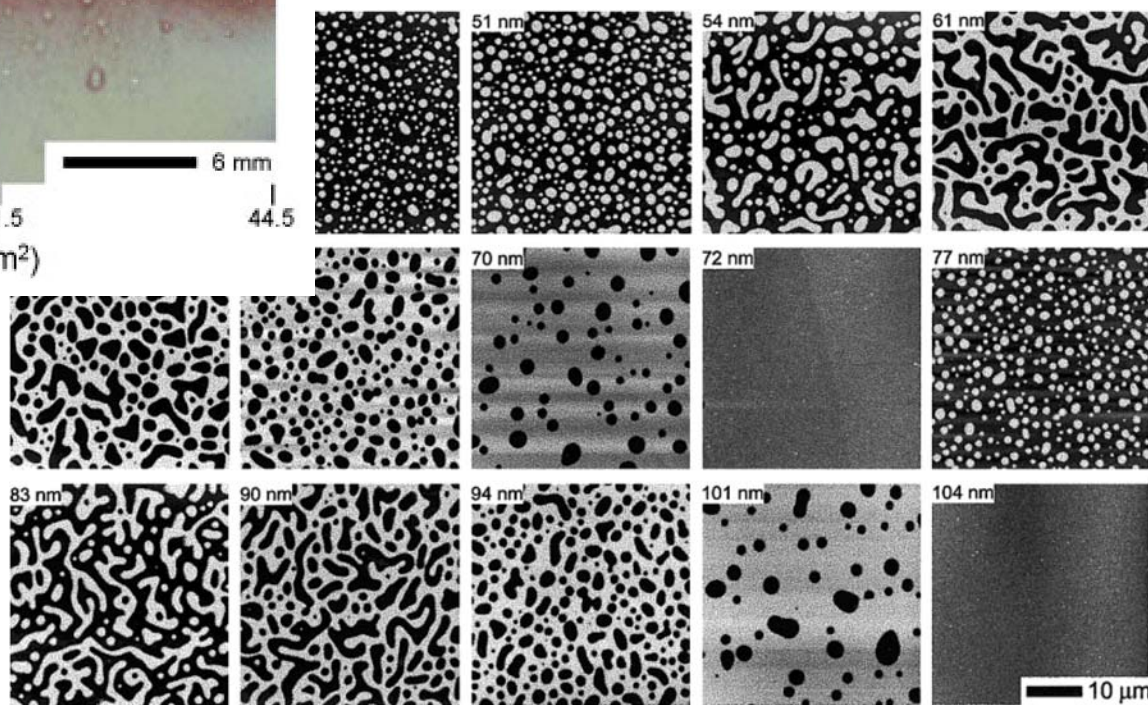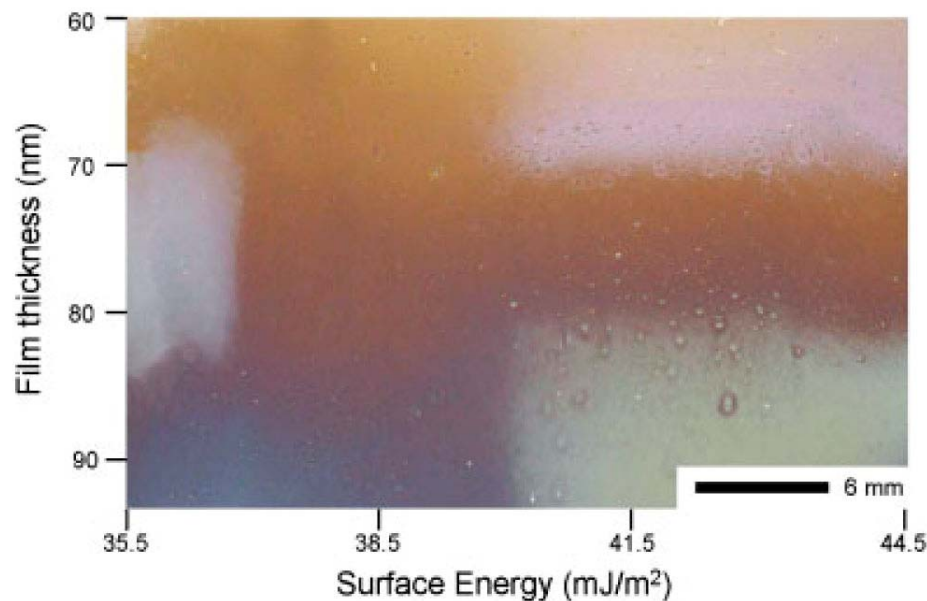
Smith et al. *J. Polym. Sci. B: Polym. Phys.* **2001**, *39*, 2141-2158.
Smith et al. *Phys. Rev. Lett.* **2001**, *87*, 015503.

Flow coating – next generation

- ❏ New stages
    - ❏ Longer range of travel (100 mm)
    - ❏ Higher velocities (63 mm/s)
    - ❏ Limit / home switches
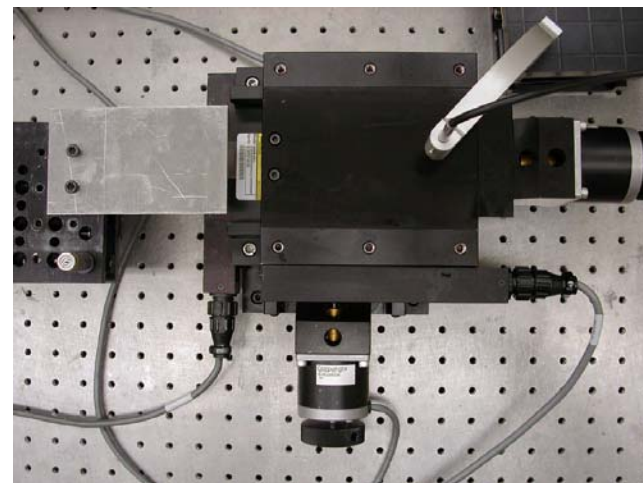   - ❏ Single motion controller

- ❏ Flow coater only requires one stage; second stage provides mapping capabilities

- ❏ Total cost - $15k
    - ❏ stages
    - ❏ motion controllers
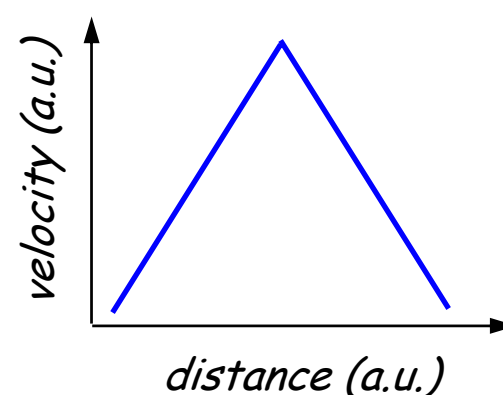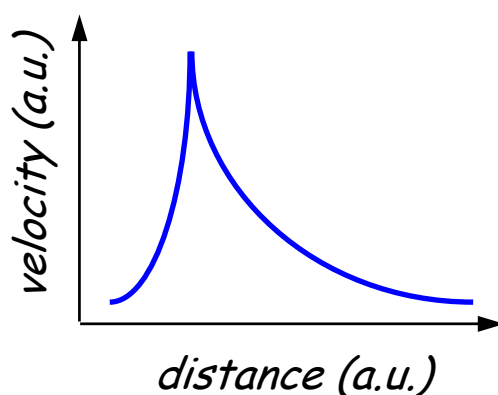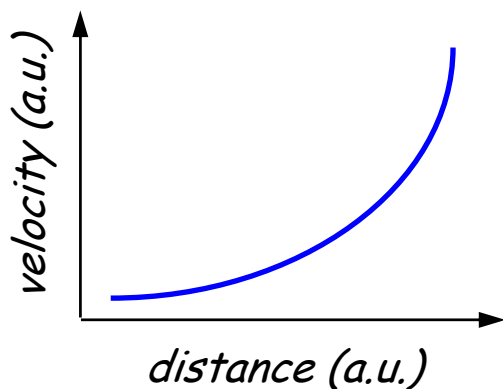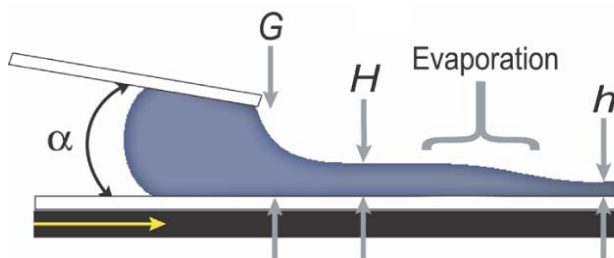   - ❏ PCI card
    - ❏ tip/tilt stage and brackets

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Future Work

- ❑ Develop model for coating flows in this geometry
- ❑ Explore upper limit of thickness regime
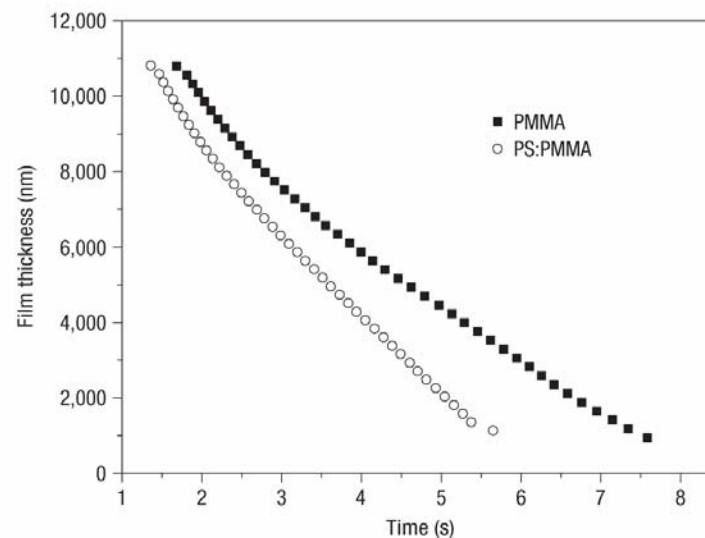- ❑ Non-linear acceleration profiles

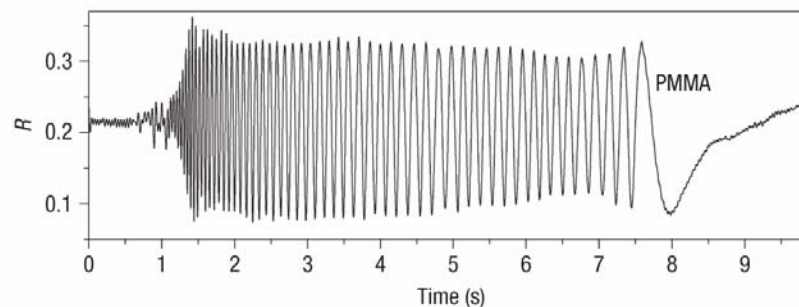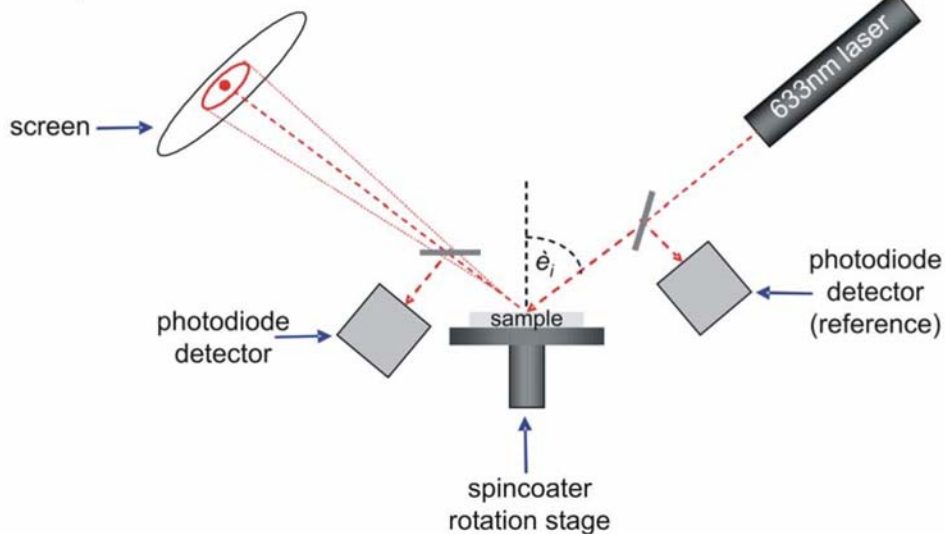- ❑ Other geometrical factors

□ We know the gap height and final film thickness.

□ But we are blind to everything that
  happens in-between.

□ Specular reflectivity -> answers.

S.Y. Heriot and R.A.L. Jones, *Nat. Mater.* **2005**, *4*, 782-786.

Conclusions

- ❑ NCMC flow coater is a versatile combinatorial tool
- ❑ Can generate thickness gradients ranging from 10 nm to 10 μm
- ❑ We have started benchmark tests
- ❑ Benchmarks will validate the coating flow model

Acknowledgments

- ❑ Wenhua Zhang & Xuesong Hu – Python motion control
- ❑ Kristen Roskov – SURF student